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Glycyl-L-alanine: a multi-temperature neutron study

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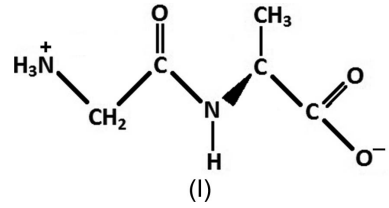
Neutron diffraction data have been collected at 12, 50, 150 and 295 K for the dipeptide glycyl-L-alanine, $C_5H_{10}N_2O_3$, in order to obtain accurate positional and anisotropic displacement parameters for the H atoms. The values of these parameters serve as a benchmark for assessing the equivalent parameters obtained from a so-called Hirshfeld-atom refinement of X-ray diffraction data described elsewhere [Capelli *et al.* (2014). *IUCrJ*, **1**, 361–379]. The flexibility of the glycyl-L-alanine molecule in the solid and the hydrogen-bonding interactions as a function of temperature are also considered.

Keywords: crystal structure; neutron diffraction; multi-temperature study; glycyl-L-alanine; dipeptide.

1. Introduction

Conformations of simple oligopeptides have been extensively studied to aid in the understanding of the secondary structure of proteins. The dipeptide glycyl-L-alanine, whose X-ray structure was first reported in 1979 (Wang & Paul, 1979), has been used as a model system for studying several phenomena, *viz.* photo-induced electron transfer (Hill *et al.*, 1996), correlation of thermal properties and solid-state behaviour (Barone & Puliti, 1999), and solvent effects on conformation and vibrational spectra (Nandini & Sathyanarayana, 2003). More recently, the flexibility of the glycylalanine chain has been used to build a MOF-like (MOF is metal–organic framework) crystalline material with adaptable pore sizes (Rabone *et al.*, 2010). In the present work, accurate positional and anisotropic displacement parameters for the H atoms have been obtained from neutron diffraction data collected at 12, 50, 150 and 295 K. The values of these parameters serve as a benchmark for assessing the positional and anisotropic displacement parameters of glycyl-L-alanine obtained from a so-called Hirshfeld-atom refinement of X-ray diffraction data, as described elsewhere (Capelli *et al.*, 2014). Also of interest are the flexibility of the glycyl-L-alanine molecule in the solid state and the hydrogen-bonding interactions as a function of temperature.



(I)

2. Experimental

2.1. Synthesis and crystallization

The title compound was purchased from Sigma–Aldrich as a powder and was recrystallized by slow evaporation from an aqueous solution.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Neutron diffraction measurements were conducted at the Institut Laue–Langevin (ILL), Grenoble, on the high-resolution four-circle diffractometer D9 equipped with a small-size position-sensitive ^{3}He-gas detector (Lehmann *et al.*, 1989) that allows an optimal discrimination of the Bragg peak from the background. A wavelength of 0.8313 (2) Å from a Cu(220) monochromator was chosen for the parametric data collection at four temperatures, *i.e.* 12, 50, 150 and 295 K.

A crystal of about 3 × 3 × 1.5 mm was sealed in a 2 K four-circle cryo-refrigerator and cooled slowly (at a rate of 2 K min^{-1}) to 12 K while monitoring a very strong reflection (600) in order to monitor crystal quality. No significant changes in the crystal mosaic spread nor splitting of peaks was observed during cooling. The space group $P2_12_12_1$, already determined by Wang & Paul (1979), was confirmed at low temperature.

Reflections were measured in equatorial geometry with ω-X-θ scans. The scan width was increased with increasing θ angle of the reflection corresponding to the resolution function of the D9 instrument. Counting times were 4–7 s per step. Reflections at very low θ angle were measured with simple ω scans. Reflections of the type $\pm h, +k, \pm l$ were measured to a maximum diffracting angle of $2\theta_{max} = 80^\circ$ at 12 K and $2\theta_{max} = 76^\circ$ at other temperatures.

Bragg intensities were integrated in three dimensions following the method of Wilkinson *et al.* (1988) as implemented in the ILL program *RACER* and corrected for the Lorentz effect. Absorption corrections were applied based on Gaussian integration (Coppens *et al.*, 1965) using as boundary planes the indexed faces of the crystal and the calculated attenuation coefficient, taking also into account the wavelength dependence of the incoherent scattering of H atoms (Howard *et al.*, 1987).

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Table 1
Experimental details.

	(I) at 12 K	(I) at 50 K	(I) at 150 K	(I) at 295 K
Crystal data				
Chemical formula	$C_5H_{10}N_2O_3$	$C_5H_{10}N_2O_3$	$C_5H_{10}N_2O_3$	$C_5H_{10}N_2O_3$
M_r	146.15	146.15	146.15	146.15
Crystal system, space group	Orthorhombic, $P2_12_12_1$	Orthorhombic, $P2_12_12_1$	Orthorhombic, $P2_12_12_1$	Orthorhombic, $P2_12_12_1$
Temperature (K)	12	50	150	295
a, b, c (Å)	7.4541 (15), 9.4918 (19), 9.7287 (19)	7.4587 (7), 9.4928 (9), 9.7250 (9)	7.4871 (16), 9.4966 (19), 9.7078 (19)	7.5302 (11), 9.5115 (16), 9.6855 (14)
V (Å^3)	688.3 (2)	688.57 (11)	690.2 (2)	693.71 (18)
Z	4	4	4	4
Radiation type	neutron, λ = 0.83130 Å	neutron, λ = 0.83130 Å	neutron, λ = 0.83130 Å	neutron, λ = 0.83130 Å
μ (mm^{-1})	2.15	2.15	2.15	2.15
Crystal size (mm)	3 × 3 × 1.5	3 × 3 × 1.5	3 × 3 × 1.5	3 × 3 × 1.5
Data collection				
Diffractometer	4-circle Eulerian cradle	4-circle Eulerian cradle	4-circle Eulerian cradle	4-circle Eulerian cradle
Absorption correction	Gaussian, ILL-DATAP software	Gaussian, ILL-DATAP software	Gaussian, ILL-DATAP software	Gaussian, ILL-DATAP software
T_{min}, T_{max}	0.726, 0.808	0.726, 0.808	0.726, 0.808	0.726, 0.808
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	2237, 1960, 1692	1467, 1354, 1205	1481, 1354, 1165	1446, 1354, 1048
R_{int}	0.029	0.024	0.024	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.774	0.740	0.740	0.742
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.106, 1.08	0.042, 0.084, 1.07	0.045, 0.081, 1.07	0.061, 0.102, 1.10
No. of reflections	1960	1354	1354	1354
No. of parameters	182	182	182	182
H-atom treatment	All H-atom parameters refined	All H-atom parameters refined	All H-atom parameters refined	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (fermion Å^{-3})	1.18, −1.28	1.03, −0.88	0.82, −0.90	0.81, −0.84

Computer programs: ILL-MAD software, ILL-RAFD9 software, *RACER* (Wilkinson *et al.*, 1988), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

The starting model for the structure refinement at 12 K was based on the atomic coordinates for the heavy atoms taken from an X-ray structure determination at the same temperature (Capelli *et al.*, 2014). The refined 12 K neutron structural model was then used as a starting model for the higher temperatures. The refinement of all structures was done by full-matrix least-squares on F^2 using *SHELXL97* (Sheldrick, 2008), using the coherent scattering amplitudes tabulated by Rauch & Waschkowski (2003). All H atoms were located in a difference Fourier map and all atoms were refined anisotropically.

3. Results and discussion

The glycyl-L-alanine molecule (Fig. 1) crystallizes in its zwitterionic form, with the ammonium group bearing three H atoms in a tetrahedral configuration at an average distance of 1.044 (2) Å from the N atom (average over all temperatures); the two carboxylate O atoms are almost equidistant from the C atom at the three lowest temperatures [on average 1.251 (2) Å for C1—O1 and 1.259 (2) Å for C1—O2]. These C1—O bond lengths become virtually indistinguishable at 295 K [1.248 (5) and 1.249 (4) Å]. The main chain of the

Table 2
Details of selected hydrogen bonds (Å, °) as a function of temperature for the structure of glycyl-L-alanine.

	T (K)	D—H	H···A	D···A	D—H···A	Symmetry code on A
N1—H1N1···O1	12	1.023 (4)	1.868 (4)	2.876 (2)	167.8 (4)	$-x+\frac{1}{2}, -y+2, z-\frac{1}{2}$
	50	1.018 (4)	1.866 (4)	2.870 (2)	168.3 (4)	
	150	1.025 (4)	1.869 (5)	2.880 (3)	168.3 (4)	
	295	1.024 (6)	1.891 (6)	2.903 (4)	169.3 (5)	
N2—H1N2···O2	12	1.044 (4)	1.711 (5)	2.747 (3)	170.8 (4)	$-x+\frac{3}{2}, -y+2, z-\frac{1}{2}$
	50	1.045 (4)	1.713 (5)	2.751 (3)	171.5 (4)	
	150	1.041 (5)	1.721 (5)	2.755 (3)	171.4 (5)	
	295	1.042 (8)	1.737 (8)	2.771 (4)	171.2 (7)	
N2—H2N2···O1	12	1.045 (4)	1.686 (5)	2.716 (2)	167.6 (4)	$x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$
	50	1.042 (4)	1.693 (5)	2.720 (2)	167.71 (4)	
	150	1.052 (5)	1.682 (5)	2.718 (3)	167.1 (5)	
	295	1.043 (7)	1.684 (7)	2.712 (4)	167.5 (7)	
N2—H3N2···O2	12	1.044 (4)	1.729 (5)	2.723 (2)	157.6 (4)	$-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$
	50	1.042 (4)	1.735 (5)	2.726 (2)	157.2 (4)	
	150	1.040 (5)	1.740 (5)	2.728 (3)	157.1 (4)	
	295	1.049 (7)	1.744 (8)	2.740 (4)	156.7 (6)	

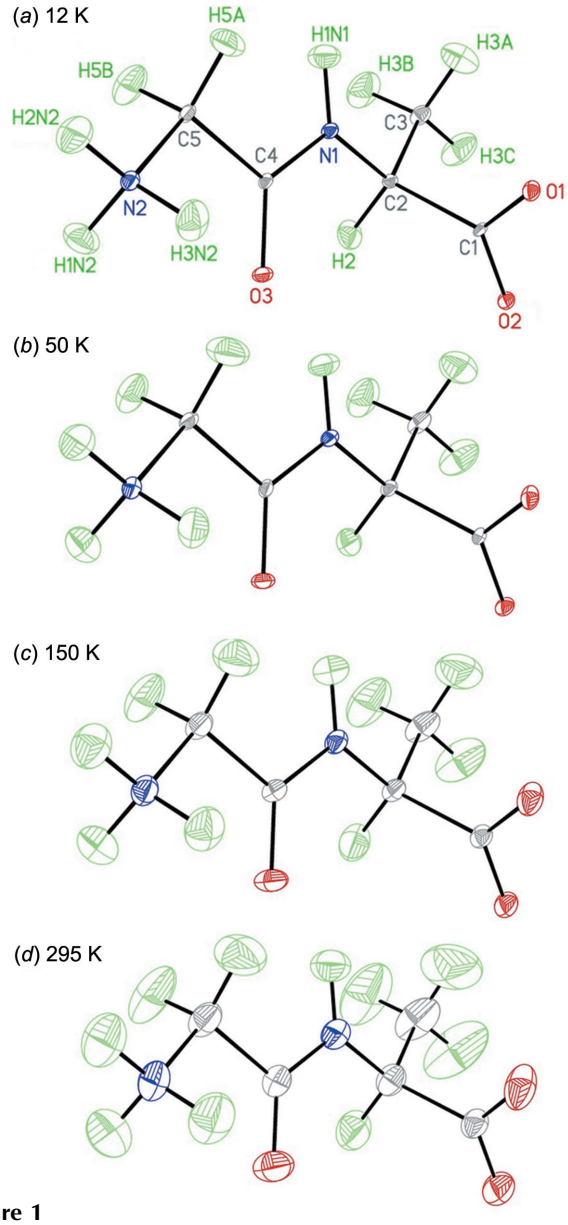

Figure 1
ORTEPIII (Burnett & Johnson, 1996) representation of the molecular structure of glycyl-L-alanine at (*a*) 12, (*b*) 50, (*c*) 150 and (*d*) 295 K, showing the atom-labelling scheme used. Displacement ellipsoids are drawn at the 50% probability level.

molecule, from H2N2—N2 to C3—H3*C*, shows an overall *trans*–planar conformation that does not change over the range of temperatures analysed. The largest deviation from planarity is associated with the C4—N1—C2—C3 torsion angle [161.63 (15)° at 12 K]. The carboxylate C atom has a *gauche* conformation with respect to the main chain, with a C4—N1—C2—C1 torsion angle of −77.12 (18)°. The conformation of the main chain allows the three groups involved in hydrogen bonding, *i.e.* NH, NH_3^+ and COO^-, to form a complex three-dimensional network with eight neighbouring molecules in the first coordination sphere (Fig. 2). Both carboxylate O atoms are acceptors in two hydrogen bonds each, *i.e.* atom O2 interacts with two H atoms of different ammonium groups, while atom O1 has interactions with an

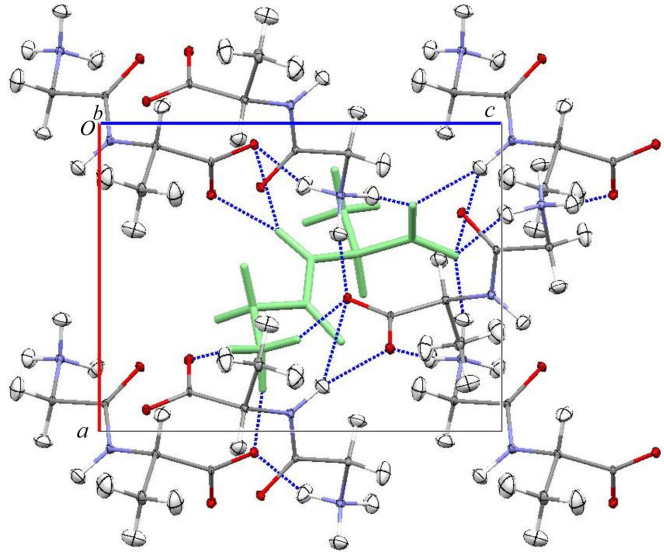

Figure 2
View down the *b* axis of the three-dimensional hydrogen-bonded network in glycyl-L-alanine at 12 K. Only the eight molecules in the first coordination sphere are shown. The central molecule is given in light green and in stick style for clarity. The blue dashed lines represent hydrogen-bond interactions.

ammonium H atom and the H atom on the peptidic N atom. The geometries of the shortest hydrogen bonds are reported in Table 2 and show no significant variations across the four temperatures.

In the reported structure of the metal–dipeptide framework $[Zn(Gly\text{-}Ala)_2]\cdot$(solvent) (Rabone *et al.*, 2010), the dipeptide molecule also adopts a *trans*–planar conformation, but with the two extremes being the ammonium N atom on one side and the carboxylate C atom on the other. The largest deviation from planarity is associated with the C4—N1—C2—C1 torsion angle (−143.5°). The methyl group is in a distorted *gauche* position relative to the N1···C1 chain. In this conformation, four dipeptide molecules can tetrahedrally coordinate a Zn atom forming a grid-like layered structure with pores that accommodate solvent molecules.

Note that the same molecular chain adopts different conformations in order to optimize the interactions with its environment; the conformation adopted in the structure of the dipeptide alone maximizes the number of hydrogen-bonding interactions in three dimensions, while the conformation in the metal–dipeptide framework permits tetrahedral coordination of the Zn counter-ion and formation of the framework structure.

A detailed discussion of the evolution of the anisotropic displacement parameters (ADPs) with temperature is beyond the scope of this report but a few considerations can be made. Preliminary calculations performed with the program *NKA* (Bürgi *et al.*, 2004) show that using a classic **TLS** rigid-body description at a single temperature (Schomaker & Trueblood, 1968), the fitting between calculated and observed ADPs deteriorates with decreasing temperature (agreement factors of 30% at 295 K, 34% at 150 K, 45% at 50 K and 48% at 12 K). This result suggest that internal motions in glycyl-L-

alanine become increasingly important compared to rigid-body translations and librations. Using the six degrees of freedom of a rigid body in a molecular Einstein model that describe the ADPs at all temperatures with a unique set of frequencies/eigenvectors and additional additive tensors accounting for the internal high-frequency motions (Bürgi & Capelli, 2000), the fitting between observed and modelled ADPs becomes more reasonable, with an agreement factor of 20%, but the tensors accounting for the high-frequency motions turn out to be much larger than expected for some atoms in specific directions. For example, the ellipsoids of the O atoms of the carboxylate group (Fig. 1) look elongated in the direction orthogonal to the O1—C1—O2 plane, suggesting a librational motion of this group around the C1—C2 direction. The diagonal components of the tensor corresponding to atoms O1 and O2 are: ε_{11} = 0.0025 (9) Å^2, ε_{22} = 0.0015 (9) Å^2, ε_{33} = 0.0070 (9) Å^2, where ε_{11} and ε_{22} represent the in-plane directions and ε_{33} the out-of-plane one. The corresponding calculated value from spectroscopic data and *ab initio* calculations for the two in-plane motions for the O atom in the carbonyl group of urea is 0.0008 Å^2 (see Table 7 in Capelli *et al.*, 2000), much smaller than the diffraction-derived values. Although the comparison is made between a carboxylate and a carbonyl group, the large discrepancy between the calculated and observed values for the high-frequency tensor of the O atoms strongly indicates that a more flexible model of motion has to be used to describe the motion of the carboxylate group, especially in the out-of-plane direction.

Analogous considerations on the orientation of ADPs can be applied to the atoms in both the methyl and the ammonium groups. In both cases, the largest elements of the high-frequency tensor point in the direction of librational motions that increase in amplitude with increasing temperature. In the case of the $-NH_3^+$ group, the motion shows a reduced amplitude compared to the $-CH_3$ group; this is most likely due to the hydrogen-bonding interactions that reduce the movements of the H atoms.

References

Barone, G. & Puliti, R. (1999). *J. Therm. Anal. Calorim.* **57**, 119–132.

Bürgi, H. B. & Capelli, S. C. (2000). *Acta Cryst.* A**56**, 403–412.

Bürgi, H. B., Förtsch, M., Capelli, S. C. & Hauser, J. (2004). *NKA. Program for Normal Coordinate Analysis from Anisotropic Displacement Parameters at Multiple Temperatures*. University of Bern, Switzerland.

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.

Capelli, S. C., Bürgi, H.-B., Dittrich, B., Grabowsky, S. & Jayatilaka, D. (2014). *IUCrJ*, **1**, 361–379.

Capelli, S. C., Förtsch, M. & Bürgi, H. B. (2000). *Acta Cryst.* A**56**, 413–424.

Coppens, P., Leiserowitz, L. & Rabinovich, D. (1965). *Acta Cryst.* **18**, 1035–1038.

Hill, R. R., Jeffs, G. E., Banaghan, F., McNally, T. & Werninck, A. R. (1996). *J. Chem. Soc. Perkin Trans. 2*, pp. 1595–1599.

Howard, J. A. K., Johnson, O., Schultz, A. J. & Stringer, A. M. (1987). *J. Appl. Cryst.* **20**, 120–122.

Lehmann, M. S., Kuhs, W. F., McIntyre, G. J., Wilkinson, C. & Allibon, J. R. (1989). *J. Appl. Cryst.* **22**, 562–568.

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.

Nandini, G. & Sathyanarayana, D. N. (2003). *J. Mol. Struct. (THEOCHEM)*, **638**, 79–90.

Rabone, J., Yue, Y.-F., Chong, S. Y., Stylianou, K. C., Bacsa, J., Bradshaw, D., Darling, G. R., Berry, N. G., Khimyak, Y. Z., Ganin, A. Y., Wiper, P., Claridge, J. B. & Rosseinsky, M. J. (2010). *Science*, **329**, 1053–1057.

Rauch, A. & Waschkowski, W. (2003). In *Neutron Data Booklet*, edited by A.-J. Dianoux & G. Lander. Philadelphia: OPC Science.

Schomaker, V. & Trueblood, K. N. (1968). *Acta Cryst.* B**24**, 63–76.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Wang, A. H. J. & Paul, I. C. (1979). *Cryst. Struct. Commun.* **8**, 269–273.

Wilkinson, C., Khamis, H. W., Stansfield, R. F. D. & McIntyre, G. J. (1988). *J. Appl. Cryst.* **21**, 471–478.

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Computing details

For all compounds, data collection: ILL-MAD software; cell refinement: ILL-RAFD9 software; data reduction: RACER (Wilkinson *et al.*, 1988); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008).

(ga12k) Glycyl-*L*-alanine

Crystal data

$C_5H_{10}N_2O_3$
M_r = 146.15
Orthorhombic, $P2_12_12_1$
a = 7.4541 (15) Å
b = 9.4918 (19) Å
c = 9.7287 (19) Å
V = 688.3 (2) Å^3
Z = 4
$F(000)$ = 128
D_x = 1.410 Mg m^{-3}
? radiation, λ = 0.83130 Å
Cell parameters from 704 reflections
θ = 3.9–39.9°
μ = 2.15 mm^{-1}
T = 12 K
Prismatic, coulourless
3 × 3 × 1.5 mm

Data collection

4-circle Eulerian cradle diffractometer
Radiation source: nuclear reactor
Cu(220) monochromator
Detector resolution: 0.5 pixels mm^{-1}
ω–X–θ scans
Absorption correction: gaussian ILL-DATAP software
T_{min} = 0.726, T_{max} = 0.808
2237 measured reflections
1960 independent reflections
1692 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 40.0°, θ_{min} = 3.5°
h = −11→6
k = 0→14
l = −15→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.106
S = 1.08
1960 reflections
182 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0194P)^2 + 7.4533P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 1.18 e Å^{-3}
$\Delta\rho_{min}$ = −1.28 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0122 (15)
Absolute structure: Flack (1983), **???? Friedel pairs**
Absolute structure parameter: −10 (10)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.
Note that the absolute structure Flack parameter is meaningless in this structure determination because in a neutron diffraction experiment there is no anomalous effect.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2682 (3)	0.9623 (2)	0.7780 (2)	0.0073 (3)
O2	0.4306 (3)	1.1255 (2)	0.8838 (2)	0.0074 (3)
O3	0.7088 (3)	0.9487 (2)	0.6001 (2)	0.0073 (3)
N1	0.44249 (17)	1.03639 (14)	0.52565 (13)	0.0057 (2)
N2	0.73423 (17)	0.75557 (14)	0.40105 (13)	0.0060 (2)
C1	0.3735 (2)	1.06534 (19)	0.77694 (17)	0.0046 (3)
C2	0.4255 (2)	1.1344 (2)	0.63936 (17)	0.0050 (3)
C3	0.2832 (3)	1.2448 (2)	0.6045 (2)	0.0091 (3)
C4	0.5884 (2)	0.95488 (19)	0.51374 (17)	0.0046 (3)
C5	0.5985 (2)	0.8671 (2)	0.38191 (18)	0.0060 (3)
H1N1	0.3532 (6)	1.0410 (5)	0.4460 (4)	0.0177 (7)
H1N2	0.8624 (6)	0.7996 (5)	0.4051 (5)	0.0188 (7)
H2N2	0.7358 (7)	0.6793 (5)	0.3236 (5)	0.0189 (8)
H3N2	0.7031 (6)	0.7006 (5)	0.4909 (5)	0.0199 (8)
H2	0.5544 (6)	1.1871 (5)	0.6555 (4)	0.0172 (7)
H3A	0.1537 (7)	1.1938 (6)	0.5847 (6)	0.0272 (10)
H3B	0.3232 (8)	1.3042 (7)	0.5132 (6)	0.0301 (11)
H3C	0.2686 (9)	1.3189 (6)	0.6882 (5)	0.0279 (11)
H5A	0.4674 (6)	0.8171 (6)	0.3591 (5)	0.0216 (9)
H5B	0.6372 (8)	0.9340 (5)	0.2943 (5)	0.0230 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0079 (7)	0.0085 (7)	0.0055 (7)	−0.0027 (7)	0.0009 (6)	−0.0015 (7)
O2	0.0069 (7)	0.0104 (8)	0.0049 (7)	−0.0015 (7)	0.0002 (7)	−0.0023 (6)
O3	0.0069 (7)	0.0099 (8)	0.0052 (7)	0.0025 (6)	−0.0019 (6)	−0.0010 (6)
N1	0.0060 (5)	0.0067 (4)	0.0045 (4)	0.0018 (4)	−0.0004 (4)	−0.0009 (4)
N2	0.0064 (5)	0.0065 (4)	0.0049 (4)	0.0009 (4)	0.0000 (4)	−0.0006 (4)
C1	0.0056 (6)	0.0058 (6)	0.0024 (6)	0.0001 (5)	−0.0002 (5)	0.0002 (5)
C2	0.0052 (6)	0.0055 (6)	0.0044 (6)	0.0007 (6)	−0.0004 (5)	0.0003 (5)
C3	0.0119 (7)	0.0083 (7)	0.0071 (7)	0.0027 (6)	−0.0004 (6)	0.0002 (6)
C4	0.0052 (6)	0.0056 (6)	0.0031 (6)	0.0011 (6)	0.0003 (5)	−0.0011 (5)
C5	0.0071 (7)	0.0075 (7)	0.0033 (6)	0.0019 (6)	0.0003 (5)	−0.0001 (5)

H1N1	0.0161 (16)	0.0212 (17)	0.0157 (15)	0.0035 (16)	−0.0053 (13)	−0.0020 (14)
H1N2	0.0114 (15)	0.0222 (18)	0.0228 (19)	−0.0025 (14)	−0.0027 (15)	−0.0014 (16)
H2N2	0.022 (2)	0.0179 (18)	0.0164 (16)	0.0006 (16)	−0.0005 (16)	−0.0069 (14)
H3N2	0.022 (2)	0.0211 (18)	0.0165 (17)	0.0052 (16)	0.0049 (15)	0.0082 (14)
H2	0.0151 (17)	0.0190 (18)	0.0174 (16)	−0.0066 (15)	0.0028 (14)	−0.0031 (14)
H3A	0.0176 (18)	0.029 (2)	0.035 (3)	0.0006 (18)	−0.006 (2)	0.000 (2)
H3B	0.038 (3)	0.031 (2)	0.021 (2)	0.005 (2)	0.001 (2)	0.0111 (19)
H3C	0.040 (3)	0.027 (2)	0.0165 (18)	0.013 (2)	−0.0008 (19)	−0.0059 (16)
H5A	0.0107 (16)	0.032 (2)	0.0223 (19)	−0.0010 (16)	−0.0032 (14)	−0.0099 (17)
H5B	0.035 (2)	0.0204 (19)	0.0133 (15)	0.0045 (17)	0.0031 (16)	0.0037 (15)

Geometric parameters (Å, º)

O1—C1	1.254 (3)	C1—C2	1.540 (3)
O2—C1	1.260 (2)	C2—C3	1.529 (3)
O3—C4	1.230 (2)	C2—H2	1.095 (5)
N1—C4	1.340 (2)	C3—H3A	1.097 (6)
N1—C2	1.451 (2)	C3—H3B	1.093 (6)
N1—H1N1	1.023 (4)	C3—H3C	1.082 (6)
N2—C5	1.476 (2)	C4—C5	1.531 (3)
N2—H1N2	1.044 (4)	C5—H5A	1.109 (5)
N2—H2N2	1.045 (5)	C5—H5B	1.102 (5)
N2—H3N2	1.044 (5)		
C4—N1—C2	120.47 (14)	C1—C2—H2	106.9 (3)
C4—N1—H1N1	119.2 (3)	C2—C3—H3A	110.3 (3)
C2—N1—H1N1	119.6 (3)	C2—C3—H3B	110.2 (4)
C5—N2—H1N2	110.2 (3)	C2—C3—H3C	110.4 (3)
C5—N2—H2N2	114.4 (3)	H3A—C3—H3B	109.0 (5)
C5—N2—H3N2	108.2 (3)	H3A—C3—H3C	109.3 (5)
H1N2—N2—H2N2	107.2 (4)	H3B—C3—H3C	107.7 (5)
H1N2—N2—H3N2	111.8 (4)	O3—C4—N1	124.08 (17)
H2N2—N2—H3N2	105.1 (4)	O3—C4—C5	120.67 (17)
O1—C1—O2	123.91 (19)	N1—C4—C5	115.25 (15)
O1—C1—C2	119.79 (17)	N2—C5—C4	108.56 (14)
O2—C1—C2	116.05 (17)	N2—C5—H5A	108.8 (3)
N1—C2—C3	109.32 (14)	C4—C5—H5A	110.9 (3)
N1—C2—C1	114.30 (15)	N2—C5—H5B	109.4 (3)
C3—C2—C1	108.06 (14)	C4—C5—H5B	110.3 (3)
N1—C2—H2	109.0 (3)	H5A—C5—H5B	108.8 (4)
C3—C2—H2	109.1 (3)		

(ga50k) Glycyl-*L*-alanine

Crystal data

$C_5H_{10}N_2O_3$
M_r = 146.15
Orthorhombic, $P2_12_12_1$
a = 7.4587 (7) Å
b = 9.4928 (9) Å
c = 9.7250 (9) Å
V = 688.57 (11) Å^3
Z = 4

$F(000) = 128$
$D_x = 1.410$ Mg m^{-3}
? radiation, $\lambda = 0.83130$ Å
Cell parameters from 553 reflections
$\theta = 4.0–37.9°$

$\mu = 2.15$ mm^{-1}
$T = 50$ K
Prismatic, coulourless
3 × 3 × 1.5 mm

Data collection

4-circle Eulerian cradle diffractometer
Radiation source: nuclear reactor
Cu(220) monochromator
Detector resolution: 0.5 pixels mm^{-1}
ω–X–θ scans
Absorption correction: gaussian ILL-DATAP software
$T_{min} = 0.726$, $T_{max} = 0.808$

1467 measured reflections
1354 independent reflections
1205 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 38.0°$, $\theta_{min} = 3.5°$
$h = -11 \rightarrow 6$
$k = 0 \rightarrow 14$
$l = -14 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.084$
$S = 1.07$
1354 reflections
182 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map

All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0199P)^2 + 3.9417P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 1.03$ e Å^{-3}
$\Delta\rho_{min} = -0.88$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0144 (15)
Absolute structure: Flack (1983), **???? Friedel pairs**
Absolute structure parameter: −10 (10)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.
Note that the absolute structure Flack parameter is meaningless in this structure determination because in a neutron diffraction experiment there is no anomalous effect.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2675 (3)	0.9627 (2)	0.7782 (2)	0.0089 (4)
O2	0.4303 (3)	1.1252 (2)	0.8836 (2)	0.0084 (4)
O3	0.7085 (3)	0.9487 (2)	0.6002 (2)	0.0085 (3)
N1	0.44236 (17)	1.03665 (14)	0.52554 (12)	0.0068 (2)
N2	0.73413 (17)	0.75581 (15)	0.40101 (13)	0.0072 (2)
C1	0.3731 (2)	1.06477 (19)	0.77695 (17)	0.0057 (3)
C2	0.4259 (3)	1.1351 (2)	0.63902 (17)	0.0065 (3)

C3	0.2834 (3)	1.2450 (2)	0.6043 (2)	0.0117 (3)
C4	0.5889 (2)	0.9549 (2)	0.51396 (17)	0.0058 (3)
C5	0.5976 (2)	0.8671 (2)	0.38213 (17)	0.0071 (3)
H1N1	0.3538 (5)	1.0398 (5)	0.4459 (4)	0.0178 (7)
H1N2	0.8619 (6)	0.8009 (5)	0.4043 (5)	0.0206 (8)
H2N2	0.7351 (6)	0.6795 (5)	0.3241 (4)	0.0192 (8)
H3N2	0.7041 (6)	0.7018 (5)	0.4914 (4)	0.0205 (8)
H2	0.5558 (6)	1.1864 (5)	0.6557 (4)	0.0190 (7)
H3A	0.1559 (7)	1.1944 (6)	0.5854 (6)	0.0315 (11)
H3B	0.3232 (8)	1.3045 (6)	0.5130 (5)	0.0329 (11)
H3C	0.2684 (9)	1.3184 (6)	0.6900 (5)	0.0325 (12)
H5A	0.4685 (6)	0.8189 (6)	0.3591 (5)	0.0248 (9)
H5B	0.6367 (7)	0.9353 (5)	0.2950 (4)	0.0234 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0101 (8)	0.0098 (8)	0.0068 (7)	−0.0042 (8)	0.0023 (7)	−0.0031 (7)
O2	0.0094 (8)	0.0108 (8)	0.0051 (7)	−0.0022 (7)	−0.0005 (7)	−0.0020 (7)
O3	0.0079 (8)	0.0107 (8)	0.0069 (7)	0.0013 (7)	−0.0030 (7)	−0.0008 (7)
N1	0.0072 (5)	0.0085 (5)	0.0048 (4)	0.0007 (4)	−0.0004 (4)	−0.0010 (4)
N2	0.0074 (5)	0.0084 (5)	0.0059 (5)	0.0007 (4)	0.0007 (4)	−0.0007 (4)
C1	0.0060 (7)	0.0074 (7)	0.0037 (6)	0.0002 (6)	0.0009 (5)	−0.0007 (6)
C2	0.0074 (7)	0.0076 (7)	0.0045 (6)	0.0009 (6)	0.0000 (6)	−0.0014 (5)
C3	0.0169 (8)	0.0108 (7)	0.0074 (7)	0.0069 (7)	−0.0004 (7)	−0.0003 (7)
C4	0.0063 (7)	0.0071 (7)	0.0039 (6)	0.0013 (6)	0.0011 (5)	−0.0003 (5)
C5	0.0077 (7)	0.0096 (7)	0.0039 (6)	0.0024 (6)	−0.0008 (6)	0.0001 (6)
H1N1	0.0170 (16)	0.0243 (18)	0.0121 (14)	0.0024 (16)	−0.0050 (12)	−0.0022 (14)
H1N2	0.0150 (17)	0.0228 (18)	0.0241 (19)	−0.0007 (15)	0.0004 (16)	0.0006 (17)
H2N2	0.0197 (19)	0.0207 (18)	0.0173 (16)	0.0010 (16)	−0.0005 (15)	−0.0054 (14)
H3N2	0.0206 (18)	0.0241 (19)	0.0166 (16)	0.0067 (16)	0.0050 (15)	0.0043 (14)
H2	0.0147 (17)	0.0255 (19)	0.0169 (15)	−0.0062 (16)	0.0014 (14)	0.0011 (15)
H3A	0.021 (2)	0.039 (3)	0.034 (2)	0.007 (2)	−0.005 (2)	0.001 (2)
H3B	0.042 (3)	0.035 (3)	0.0220 (19)	0.010 (2)	0.005 (2)	0.0106 (19)
H3C	0.049 (3)	0.027 (2)	0.021 (2)	0.014 (2)	−0.002 (2)	−0.0083 (18)
H5A	0.0175 (19)	0.029 (2)	0.028 (2)	0.0026 (17)	−0.0085 (16)	−0.0111 (18)
H5B	0.032 (2)	0.025 (2)	0.0135 (15)	0.0059 (18)	0.0039 (15)	0.0034 (15)

Geometric parameters (Å, º)

O1—C1	1.249 (3)	C1—C2	1.549 (2)
O2—C1	1.259 (3)	C2—C3	1.527 (3)
O3—C4	1.226 (3)	C2—H2	1.096 (5)
N1—C4	1.345 (2)	C3—H3A	1.081 (6)
N1—C2	1.451 (2)	C3—H3B	1.093 (6)
N1—H1N1	1.018 (4)	C3—H3C	1.092 (5)
N2—C5	1.478 (2)	C4—C5	1.531 (3)
N2—H1N2	1.045 (4)	C5—H5A	1.090 (5)

N2—H2N2	1.042 (4)	C5—H5B	1.106 (5)
N2—H3N2	1.042 (4)		
C4—N1—C2	120.26 (14)	C1—C2—H2	106.7 (3)
C4—N1—H1N1	118.7 (3)	C2—C3—H3A	110.3 (4)
C2—N1—H1N1	120.3 (3)	C2—C3—H3B	110.1 (3)
C5—N2—H1N2	109.8 (3)	C2—C3—H3C	109.8 (3)
C5—N2—H2N2	114.4 (3)	H3A—C3—H3B	109.3 (5)
C5—N2—H3N2	107.9 (3)	H3A—C3—H3C	108.8 (5)
H1N2—N2—H2N2	107.5 (4)	H3B—C3—H3C	108.6 (5)
H1N2—N2—H3N2	111.8 (4)	O3—C4—N1	124.16 (18)
H2N2—N2—H3N2	105.4 (4)	O3—C4—C5	121.07 (17)
O1—C1—O2	124.0 (2)	N1—C4—C5	114.76 (15)
O1—C1—C2	120.19 (17)	N2—C5—C4	108.32 (13)
O2—C1—C2	115.49 (17)	N2—C5—H5A	109.5 (3)
N1—C2—C3	109.31 (14)	C4—C5—H5A	111.3 (3)
N1—C2—C1	113.71 (15)	N2—C5—H5B	109.4 (3)
C3—C2—C1	107.99 (15)	C4—C5—H5B	109.5 (3)
N1—C2—H2	108.9 (3)	H5A—C5—H5B	108.7 (4)
C3—C2—H2	110.2 (3)		

(ga150k) Glycyl-*L*-alanine

Crystal data

$C_5H_{10}N_2O_3$
M_r = 146.15
Orthorhombic, $P2_12_12_1$
a = 7.4871 (16) Å
b = 9.4966 (19) Å
c = 9.7078 (19) Å
V = 690.2 (2) Å^3
Z = 4
$F(000)$ = 128

D_x = 1.406 Mg m^{-3}
? radiation, λ = 0.83130 Å
Cell parameters from 483 reflections
θ = 2.5–37.9°
μ = 2.15 mm^{-1}
T = 150 K
Prismatic, coulourless
3 × 3 × 1.5 mm

Data collection

4-circle Eulerian cradle diffractometer
Radiation source: nuclear reactor
Cu(220) monochromator
Detector resolution: 0.5 pixels mm^{-1}
ω–X–θ scans
Absorption correction: gaussian ILL-DATAP software
T_{min} = 0.726, T_{max} = 0.808

1481 measured reflections
1354 independent reflections
1165 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 38.0°, θ_{min} = 3.5°
h = −11→6
k = 0→14
l = −14→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.081
S = 1.07
1354 reflections
182 parameters

0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.P)^2 + 3.3733P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.82$ e Å^{-3}
$\Delta\rho_{min} = -0.90$ e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0143 (13)
Absolute structure: Flack (1983), **???? Friedel pairs**
Absolute structure parameter: 10 (10)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.
Note that the absolute structure Flack parameter is meaningless in this structure determination because in a neutron diffraction experiment there is no anomalous effect.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2685 (3)	0.9632 (3)	0.7778 (2)	0.0190 (4)
O2	0.4307 (3)	1.1258 (3)	0.8833 (2)	0.0161 (4)
O3	0.7094 (3)	0.9487 (3)	0.5998 (2)	0.0169 (4)
N1	0.44343 (18)	1.03566 (15)	0.52530 (13)	0.0129 (2)
N2	0.73433 (19)	0.75565 (16)	0.40072 (14)	0.0140 (3)
C1	0.3737 (3)	1.0653 (2)	0.77665 (19)	0.0114 (3)
C2	0.4268 (3)	1.1342 (2)	0.63882 (18)	0.0127 (3)
C3	0.2864 (4)	1.2446 (3)	0.6031 (2)	0.0245 (5)
C4	0.5895 (2)	0.9549 (2)	0.51366 (18)	0.0110 (3)
C5	0.5993 (3)	0.8664 (2)	0.38158 (19)	0.0141 (4)
H1N1	0.3541 (6)	1.0398 (5)	0.4454 (4)	0.0246 (8)
H1N2	0.8612 (6)	0.8002 (5)	0.4042 (5)	0.0270 (9)
H2N2	0.7358 (7)	0.6789 (5)	0.3225 (5)	0.0274 (9)
H3N2	0.7041 (7)	0.7018 (5)	0.4911 (5)	0.0270 (9)
H2	0.5565 (7)	1.1865 (6)	0.6550 (5)	0.0297 (10)
H3A	0.1592 (9)	1.1941 (8)	0.5811 (7)	0.0489 (16)
H3B	0.3250 (12)	1.3029 (8)	0.5130 (7)	0.0540 (18)
H3C	0.2720 (12)	1.3187 (7)	0.6881 (6)	0.0522 (18)
H5A	0.4706 (7)	0.8202 (6)	0.3561 (6)	0.0347 (11)
H5B	0.6362 (8)	0.9340 (6)	0.2940 (5)	0.0347 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0226 (11)	0.0208 (10)	0.0135 (8)	−0.0072 (10)	0.0042 (8)	−0.0050 (9)
O2	0.0162 (9)	0.0216 (10)	0.0104 (9)	−0.0036 (9)	0.0008 (8)	−0.0030 (8)
O3	0.0176 (9)	0.0186 (10)	0.0146 (8)	0.0027 (9)	−0.0067 (8)	−0.0023 (9)
N1	0.0136 (6)	0.0158 (6)	0.0092 (5)	0.0027 (5)	0.0006 (5)	−0.0021 (5)

N2	0.0155 (6)	0.0151 (6)	0.0114 (5)	0.0022 (5)	0.0013 (5)	−0.0010 (5)
C1	0.0131 (8)	0.0120 (8)	0.0091 (7)	0.0006 (6)	0.0007 (6)	−0.0021 (6)
C2	0.0154 (8)	0.0134 (8)	0.0094 (7)	0.0014 (7)	−0.0003 (7)	−0.0007 (6)
C3	0.0372 (13)	0.0204 (9)	0.0159 (9)	0.0145 (10)	−0.0027 (9)	0.0001 (8)
C4	0.0134 (7)	0.0109 (7)	0.0089 (6)	0.0007 (6)	−0.0012 (6)	−0.0004 (6)
C5	0.0173 (9)	0.0152 (8)	0.0099 (7)	0.0039 (7)	−0.0003 (7)	−0.0007 (7)
H1N1	0.0250 (18)	0.0282 (19)	0.0207 (16)	−0.0009 (19)	−0.0046 (14)	−0.0014 (16)
H1N2	0.0201 (19)	0.028 (2)	0.033 (2)	−0.0006 (17)	−0.0014 (18)	0.0007 (19)
H2N2	0.031 (2)	0.027 (2)	0.0243 (19)	−0.0033 (19)	0.0028 (18)	−0.0040 (17)
H3N2	0.031 (2)	0.027 (2)	0.0229 (18)	0.0060 (18)	0.0031 (17)	0.0073 (16)
H2	0.033 (2)	0.032 (2)	0.0233 (18)	−0.009 (2)	0.0048 (19)	−0.0028 (17)
H3A	0.033 (3)	0.066 (4)	0.047 (3)	0.015 (3)	−0.012 (3)	−0.002 (3)
H3B	0.083 (5)	0.046 (3)	0.034 (3)	0.016 (4)	0.005 (3)	0.015 (3)
H3C	0.079 (5)	0.042 (3)	0.035 (3)	0.032 (4)	−0.005 (3)	−0.012 (2)
H5A	0.026 (2)	0.041 (3)	0.038 (3)	0.001 (2)	−0.006 (2)	−0.013 (2)
H5B	0.056 (3)	0.029 (2)	0.0192 (17)	0.012 (2)	0.005 (2)	0.0044 (17)

Geometric parameters (Å, º)

O1—C1	1.249 (3)	C1—C2	1.542 (3)
O2—C1	1.259 (3)	C2—C3	1.525 (3)
O3—C4	1.229 (3)	C2—H2	1.102 (6)
N1—C4	1.340 (2)	C3—H3A	1.088 (8)
N1—C2	1.451 (2)	C3—H3B	1.075 (7)
N1—H1N1	1.025 (4)	C3—H3C	1.090 (6)
N2—C5	1.471 (2)	C4—C5	1.535 (3)
N2—H1N2	1.041 (5)	C5—H5A	1.088 (6)
N2—H2N2	1.052 (5)	C5—H5B	1.101 (5)
N2—H3N2	1.040 (5)		
C4—N1—C2	120.20 (15)	C1—C2—H2	107.2 (3)
C4—N1—H1N1	119.4 (3)	C2—C3—H3A	110.2 (4)
C2—N1—H1N1	119.6 (3)	C2—C3—H3B	110.7 (5)
C5—N2—H1N2	109.9 (3)	C2—C3—H3C	109.9 (4)
C5—N2—H2N2	114.3 (3)	H3A—C3—H3B	107.7 (6)
C5—N2—H3N2	108.0 (3)	H3A—C3—H3C	110.3 (7)
H1N2—N2—H2N2	107.2 (4)	H3B—C3—H3C	108.1 (6)
H1N2—N2—H3N2	111.8 (4)	O3—C4—N1	124.47 (19)
H2N2—N2—H3N2	105.7 (4)	O3—C4—C5	120.51 (19)
O1—C1—O2	124.1 (2)	N1—C4—C5	115.02 (16)
O1—C1—C2	119.96 (19)	N2—C5—C4	108.61 (15)
O2—C1—C2	115.64 (19)	N2—C5—H5A	110.4 (4)
N1—C2—C3	109.27 (16)	C4—C5—H5A	111.6 (3)
N1—C2—C1	114.07 (16)	N2—C5—H5B	110.0 (3)
C3—C2—C1	108.15 (17)	C4—C5—H5B	109.7 (3)
N1—C2—H2	108.9 (3)	H5A—C5—H5B	106.4 (5)
C3—C2—H2	109.2 (4)		

(ga295k) Glycyl-*L*-alanine

Crystal data

$C_5H_{10}N_2O_3$
M_r = 146.15
Orthorhombic, $P2_12_12_1$
a = 7.5302 (11) Å
b = 9.5115 (16) Å
c = 9.6855 (14) Å
V = 693.71 (18) Å^3
Z = 4
F(000) = 128
D_x = 1.399 Mg m^{-3}
? radiation, λ = 0.83130 Å
Cell parameters from 344 reflections
θ = 4.1–32.1°
μ = 2.15 mm^{-1}
T = 295 K
Prismatic, coulourless
3 × 3 × 1.5 mm

Data collection

4-circle Eulerian cradle diffractometer
Radiation source: nuclear reactor
Cu(220) monochromator
Detector resolution: 0.5 pixels mm^{-1}
ω–X–θ scans
Absorption correction: gaussian ILL-DATAP software
T_{min} = 0.726, T_{max} = 0.808
1446 measured reflections
1354 independent reflections
1048 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 38.1°, θ_{min} = 3.5°
h = −11→6
k = 0→14
l = −14→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.102
S = 1.10
1354 reflections
182 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.P)^2 + 3.1711P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.81 e Å^{-3}
$\Delta\rho_{min}$ = −0.84 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0147 (18)
Absolute structure: Flack (1983), **???? Friedel pairs**
Absolute structure parameter: 10 (10)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.
Note that the absolute structure Flack parameter is meaningless in this structure determination because in a neutron diffraction experiment there is no anomalous effect.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.2697 (5)	0.9635 (4)	0.7761 (3)	0.0341 (8)

O2	0.4304 (5)	1.1246 (4)	0.8829 (3)	0.0307 (7)
O3	0.7100 (5)	0.9498 (4)	0.5993 (3)	0.0305 (7)
N1	0.4455 (3)	1.0351 (2)	0.52485 (19)	0.0231 (4)
N2	0.7346 (3)	0.7560 (2)	0.4006 (2)	0.0239 (4)
C1	0.3741 (4)	1.0654 (3)	0.7764 (3)	0.0199 (5)
C2	0.4280 (4)	1.1330 (3)	0.6384 (3)	0.0225 (5)
C3	0.2895 (7)	1.2442 (4)	0.6014 (4)	0.0461 (10)
C4	0.5908 (3)	0.9548 (3)	0.5136 (2)	0.0204 (5)
C5	0.6012 (4)	0.8666 (3)	0.3813 (3)	0.0240 (6)
H1N1	0.3567 (8)	1.0374 (7)	0.4449 (6)	0.0346 (12)
H1N2	0.8614 (9)	0.7997 (8)	0.4041 (8)	0.0417 (15)
H2N2	0.7370 (10)	0.6793 (7)	0.3236 (7)	0.0391 (15)
H3N2	0.7045 (10)	0.7030 (7)	0.4927 (7)	0.0412 (15)
H2	0.5573 (10)	1.1836 (8)	0.6551 (7)	0.0438 (16)
H3A	0.1652 (15)	1.1932 (14)	0.5793 (13)	0.084 (4)
H3B	0.326 (2)	1.2995 (12)	0.5092 (10)	0.087 (4)
H3C	0.271 (2)	1.3154 (11)	0.6876 (10)	0.091 (4)
H5A	0.4733 (10)	0.8196 (9)	0.3566 (8)	0.0502 (19)
H5B	0.6412 (13)	0.9336 (9)	0.2951 (7)	0.055 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.045 (2)	0.0338 (17)	0.0239 (13)	−0.0131 (19)	0.0113 (15)	−0.0094 (14)
O2	0.0320 (16)	0.0401 (18)	0.0201 (13)	−0.0040 (16)	0.0009 (13)	−0.0059 (13)
O3	0.0321 (16)	0.0343 (17)	0.0250 (13)	0.0070 (15)	−0.0081 (13)	−0.0050 (15)
N1	0.0237 (9)	0.0282 (9)	0.0172 (7)	0.0016 (8)	0.0007 (7)	−0.0032 (7)
N2	0.0277 (10)	0.0244 (9)	0.0197 (8)	0.0018 (9)	0.0048 (8)	−0.0027 (8)
C1	0.0224 (11)	0.0218 (12)	0.0154 (10)	0.0009 (10)	0.0017 (9)	−0.0031 (9)
C2	0.0287 (13)	0.0193 (12)	0.0194 (10)	0.0020 (12)	0.0018 (11)	−0.0010 (9)
C3	0.071 (3)	0.0358 (17)	0.0318 (16)	0.031 (2)	−0.0021 (19)	0.0012 (15)
C4	0.0229 (11)	0.0213 (12)	0.0171 (9)	−0.0002 (10)	0.0018 (9)	0.0011 (9)
C5	0.0302 (14)	0.0255 (13)	0.0163 (10)	0.0046 (12)	0.0002 (10)	−0.0025 (10)
H1N1	0.030 (3)	0.042 (3)	0.031 (2)	0.007 (3)	−0.008 (2)	−0.006 (3)
H1N2	0.034 (3)	0.042 (3)	0.049 (4)	0.006 (3)	0.002 (3)	0.000 (3)
H2N2	0.049 (4)	0.034 (3)	0.034 (3)	0.004 (3)	0.006 (3)	−0.007 (2)
H3N2	0.050 (4)	0.041 (3)	0.032 (3)	0.017 (3)	0.004 (3)	0.007 (3)
H2	0.042 (4)	0.057 (4)	0.032 (3)	−0.022 (3)	0.004 (3)	−0.004 (3)
H3A	0.059 (5)	0.105 (9)	0.087 (7)	0.040 (6)	−0.022 (6)	−0.011 (7)
H3B	0.130 (10)	0.076 (7)	0.055 (5)	0.043 (7)	0.014 (6)	0.030 (5)
H3C	0.157 (12)	0.065 (6)	0.053 (5)	0.067 (7)	−0.008 (6)	−0.021 (4)
H5A	0.036 (3)	0.061 (5)	0.054 (4)	0.003 (3)	−0.010 (3)	−0.030 (4)
H5B	0.092 (6)	0.047 (4)	0.025 (2)	0.016 (4)	0.009 (3)	0.005 (3)

Geometric parameters (Å, º)

O1—C1	1.248 (5)	C1—C2	1.538 (4)
O2—C1	1.249 (4)	C2—C3	1.528 (5)

O3—C4	1.224 (4)	C2—H2	1.098 (8)
N1—C4	1.339 (3)	C3—H3A	1.076 (15)
N1—C2	1.447 (3)	C3—H3B	1.072 (11)
N1—H1N1	1.024 (6)	C3—H3C	1.084 (9)
N2—C5	1.466 (4)	C4—C5	1.533 (4)
N2—H1N2	1.042 (8)	C5—H5A	1.089 (8)
N2—H2N2	1.043 (7)	C5—H5B	1.093 (8)
N2—H3N2	1.049 (7)		

C4—O3—N1	29.12 (16)	C5—N2—H5B	29.5 (3)
C4—N1—C2	120.2 (2)	C4—N2—H5B	56.3 (2)
C4—N1—O3	26.41 (15)	H1N2—N2—H5B	90.2 (5)
C2—N1—O3	93.92 (18)	H2N2—N2—H5B	102.8 (5)
C4—N1—C5	34.97 (14)	H3N2—N2—H5B	136.8 (5)
C2—N1—C5	154.87 (19)	H5A—N2—H5B	49.8 (4)
O3—N1—C5	61.39 (12)	O1—C1—O2	124.5 (3)
C4—N1—C3	152.7 (2)	O1—C1—C2	119.3 (3)
C2—N1—C3	36.51 (16)	O2—C1—C2	116.1 (3)
O3—N1—C3	128.41 (17)	O1—C1—N1	92.5 (2)
C5—N1—C3	162.09 (15)	O2—C1—N1	141.2 (2)
C4—N1—C1	108.64 (17)	C2—C1—N1	31.58 (13)
C2—N1—C1	33.82 (14)	N1—C2—C3	109.2 (2)
O3—N1—C1	85.45 (12)	N1—C2—C1	114.6 (2)
C5—N1—C1	137.37 (13)	C3—C2—C1	108.2 (3)
C3—N1—C1	60.38 (12)	N1—C2—H2	108.2 (4)
C4—N1—H1N1	119.0 (4)	C3—C2—H2	109.6 (5)
C2—N1—H1N1	120.1 (4)	C1—C2—H2	106.8 (4)
O3—N1—H1N1	145.3 (4)	C2—C3—N1	34.29 (13)
C5—N1—H1N1	84.0 (4)	C2—C3—H3A	109.2 (7)
C3—N1—H1N1	84.1 (4)	N1—C3—H3A	89.5 (6)
C1—N1—H1N1	126.3 (4)	C2—C3—H3B	111.1 (7)
C4—N1—H2	96.1 (3)	N1—C3—H3B	91.3 (6)
C2—N1—H2	30.2 (2)	H3A—C3—H3B	106.2 (11)
O3—N1—H2	72.2 (3)	C2—C3—H3C	109.8 (7)
C5—N1—H2	127.1 (3)	N1—C3—H3C	144.1 (6)
C3—N1—H2	56.8 (3)	H3A—C3—H3C	108.8 (11)
C1—N1—H2	54.4 (2)	H3B—C3—H3C	111.5 (9)
H1N1—N1—H2	135.4 (5)	O3—C4—N1	124.5 (3)
C5—N2—C4	36.55 (13)	O3—C4—C5	120.5 (3)
C5—N2—H1N2	110.3 (4)	N1—C4—C5	115.0 (2)
C4—N2—H1N2	94.8 (4)	O3—C4—N2	87.1 (2)
C5—N2—H2N2	115.0 (5)	N1—C4—N2	147.4 (2)
C4—N2—H2N2	150.6 (4)	C5—C4—N2	34.71 (13)
H1N2—N2—H2N2	106.6 (6)	N2—C5—C4	108.7 (2)
C5—N2—H3N2	107.7 (4)	N2—C5—N1	137.11 (18)
C4—N2—H3N2	84.0 (4)	C4—C5—N1	30.04 (13)
H1N2—N2—H3N2	111.2 (6)	N2—C5—H5A	109.9 (5)
H2N2—N2—H3N2	106.0 (6)	C4—C5—H5A	111.3 (4)

C5—N2—H5A	29.1 (3)	N1—C5—H5A	88.3 (4)
C4—N2—H5A	56.8 (2)	N2—C5—H5B	109.1 (5)
H1N2—N2—H5A	138.6 (5)	C4—C5—H5B	109.5 (5)
H2N2—N2—H5A	94.2 (5)	N1—C5—H5B	100.7 (5)
H3N2—N2—H5A	96.2 (5)	H5A—C5—H5B	108.3 (7)